

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Charles M. Lieber et al.
Serial No.: 10/588,833
Confirmation No.: 4453
Filed: August 9, 2006
For: NANOSTRUCTURES CONTAINING METAL-SEMICONDUCTOR
COMPOUNDS
Examiner: D.A. Wolverton
Art Unit: 2813

**DECLARATION OF PRIOR INVENTION IN THE UNITED STATES
TO OVERCOME CITED PATENT OR PUBLICATION UNDER 37 C.F.R. §1.131**

I, Yue Wu, declare that:

1. This declaration is to establish conception and reduction to practice of the invention as recited in at least claims 126-134 and 137-145 prior to July 28, 2003, which is the effective date of Hareland, as discussed below.
2. I received my B.S. degree in Chemistry from the University of Science and Technology of China in 2001, and my Ph.D. degree in chemistry from Harvard University in 2006. I was the Miller Research Fellow in Chemistry from 2006 to 2009 at the University of California, Berkeley. I am now an assistant professor of chemical engineering at Purdue University.
3. I am an inventor of the above-identified application, and of at least claims 126-134 and 137-145.
4. I have a financial interest in the issuance of this application as a patent via at least the following. A portion of any royalties derived from licensing of the intellectual property represented by this patent application will flow to me.
5. I have read and understood the specification of the instant patent application, the pending claims, and the office action issued on June 12, 2009 (the "Office Action"), including Hareland, *et al.*, U.S. Pat. No. 6,897,098 ("Hareland").
6. The relevant date for purposes of this Declaration is the filing date of Hareland, which is July 28, 2003.

7. The conception and reduction to practice of at least claims 126-134 and 137-145 took place on a date prior to July 28, 2003. As evidence, submitted herewith as Exhibit A is a copy of a page of a laboratory notebook used by me in the regular course of my studies. The notebook page contains copies of slides I prepared that summarize experiments I designed. The date these slides were entered into my notebook has been redacted and is prior to July 28, 2003. Other slides appearing on these pages, not necessary or relevant for this Declaration, have been redacted as well. Also on these pages are example photomicrographs illustrating nanowires prepared using this method. Briefly, as shown in these slides, silicon nanowires having a diameter of 10 nm or 20 nm were positioned on a substrate and a photoresist mask was coated on top of the silicon nanowires. Nickel, a transition metal, was then evaporated onto the photoresist mask. Due to the mask, some portions of the silicon nanowires were not coated with nickel, while other portions were not covered by the mask and were consequently coated with nickel. The wires were then annealed to allow the nickel to diffuse into the silicon nanowires. The mask and excess nickel were then removed.

8. Submitted herewith as Exhibit B is a copy of another page of this laboratory notebook, containing copies of slides I prepared summarizing various additional experiments I designed and performed. These experiments were similar to the ones described in Exhibit A. The date these slides were entered into my notebook has been redacted and is prior to July 28, 2003. Other slides appearing on these pages, not necessary or relevant for this Declaration, have been redacted as well. One of these slides is labeled "EDX analysis." EDX stands for "energy-dispersive X-ray spectroscopy," and is a commonly-used analytic technique used for, among other things, determining the atomic composition of matter. As shown on this slide, I determined the ratio of nickel to silicon in the nanowires I produced to be 1.08:1 which, after accounting for experimental error (e.g., nickel residue after etching), allowed me to conclude that the nanowires contained a 1:1 stoichiometric ratio of Ni to Si and that the composition of the nanowires was therefore NiSi. Other slides in Exhibit B, each labeled "TEM Image," illustrate various zone axes of these nanowires (specifically, the $[10\bar{2}]$, $[2\bar{1}0]$, and $[01\bar{1}]$ axes), which allowed me to conclude that the NiSi portions of the nanowires were single crystals. TEM stands for "transmission electron microscopy," and is also a commonly-used analytic technique.

9. Submitted herewith as Exhibit C is a copy of another page of this laboratory notebook, containing copies of slides I prepared summarizing various additional experiments I designed and performed. In particular, these slides illustrate the results of additional testing I performed using the nanowires. The date these slides were entered into my notebook has been redacted and is prior to July 28, 2003. Other slides appearing on these pages, not necessary or relevant for this Declaration, have been redacted as well. The upper slide describes experiments where I measured resistances per unit length of $126 \Omega/\mu\text{m}$ and $131 \Omega/\mu\text{m}$ for 40 nm diameter NiSi nanowires. These can be converted to resistivities using the formula $\rho = R \frac{A}{l}$, where ρ is the resistivity, R is the resistance, A is the area, and l is the length of the nanowire. In this case, the area A of a circle is simply $\pi \left(\frac{d}{2}\right)^2$, where d is the diameter, so that the resistivities for these wires were roughly $15 \mu\Omega \text{ cm}$ or about $16 \mu\Omega \text{ cm}$, respectively. Similarly, for a 20 nm diameter NiSi nanowire, the resistance per unit length was $476 \Omega/\mu\text{m}$ and the resistivity was $15 \mu\Omega \text{ cm}$.

The lower slide illustrates an experiment where I determined the current density that a NiSi nanowire I produced could carry without failing. As shown on this slide, I measured a current density of approximately 10^8 A/cm².

10. Accordingly, as demonstrated by the notebook pages presented in Exhibits A-C, the conception and reduction to practice of at least claims 126-134 and 137-145 occurred prior to July 28, 2003, which is the filing date of Hareland.

11. All statements made herein of my own knowledge are true, and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like made in a Declaration are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-identified application or any patent issued thereon.

Date: _____

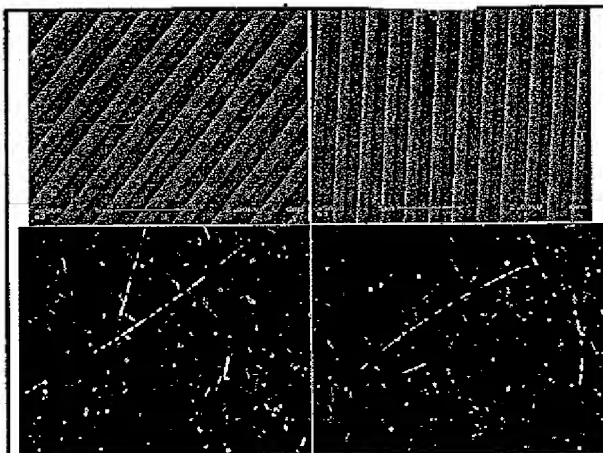
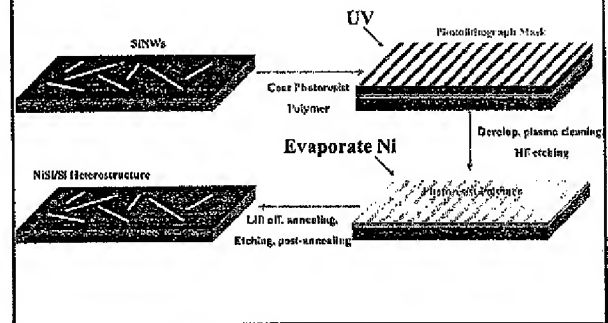
10/13/2009


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EXHIBIT A

General Approach to Achieve Heterostructure by Photolithography Method



Experiment Detail

1. NWs with Diameter of 10nm and 20nm (Intrinsic and 8000:1 p-doped) spread out on SiO₂ wafer (ZrO₂ can be dissolved in TFG)
2. Using 1805 photoresist, thickness of photoresist 400-500nm
3. Photomask 1um line with 1um spacing
4. Evaporate Ni after photolithography/develop, anneal in CVD with H₂ at 550 C for 30 min, etch Ni away by TFG at 50C for 30min, check SEM, post anneal in H₂ at 600C for 40min
5. Use wafer with high concentration of NWs for TEM (sonicate NWs off into IPA), use wafer with low concentration of NWs for transport (p-type NWs with heterostructure can be used for demonstration of integrated FETs on one NW and intrinsic NWs with heterostructure can be used for the study of ballistic transport/ SET)

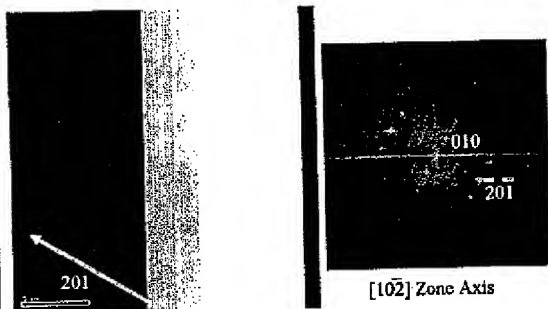
EXHIBIT B

Experiment Condition

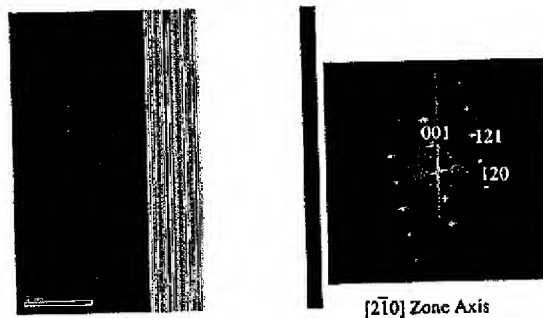
20nm Intrinsic Si nanowires were grown on substrate in H_2 at 440 degree, then quickly transferred to e-beam evaporator. 30nm Ni was evaporated on the Si nanowire growth substrate. The substrate was transferred back to the CVD setup and annealed at 550 degree for 30 minutes in H_2 . Extra Ni was removed by TFG(Ni etching solution) for 30 minutes at 50 degree, then the substrate was washed with distilled water and IPA for many times to remove the residual etching solution. The substrate was put back to CVD setup and annealed at 600 degree for 30 minutes in H_2 .

Contact on NiSi nanowires are made by 40nm Cr and 30nm Au. Before making contact, the contact area on the nanowires was oxygen plasma cleaned for 30s at 25W, then etched in HF for 5s to remove the oxide and amorphous layer. After making contact, the contact was annealed in H_2 for 3 minutes at 350 degree by RTA.

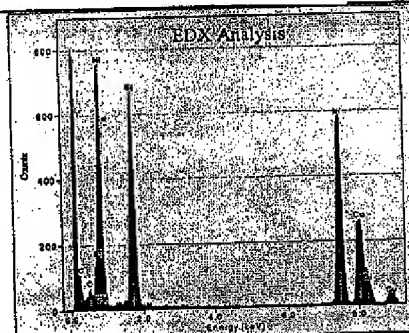
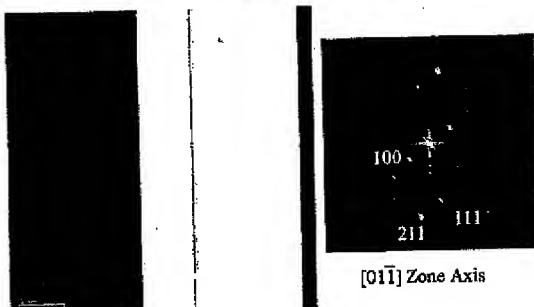
TEM Image (Nanowire grown using 10nm gold colloid)



TEM Image (Nanowire grown using 10nm gold colloid)



TEM Image (Nanowire grown using 20nm gold colloid)



Element	Wt%	At%	Wt%	At%	Wt%	At%
Si	99.99	99.99	0.01	0.01	0.01	0.01
Ni	0.01	0.01	99.99	99.99	0.01	0.01
Si	99.99	99.99	0.01	0.01	0.01	0.01
Ni	0.01	0.01	99.99	99.99	0.01	0.01
Si	99.99	99.99	0.01	0.01	0.01	0.01
Ni	0.01	0.01	99.99	99.99	0.01	0.01

Ni:Si=1.08:1

Extra Ni might come from the residual after etching.

EXHIBIT C

